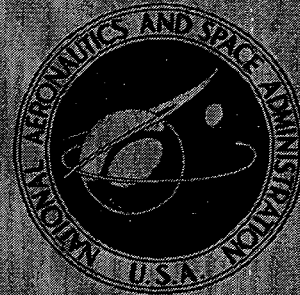


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PROCEDURE FOR RAPID DETERMINATION
OF NICKEL, COBALT, AND CHROMIUM
IN AIRBORNE PARTICULATE SAMPLES

by Warren F. Davis and Judson W. Graab

Lewis Research Center

Cleveland, Ohio 44135

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • NOVEMBER 1972

1. Report No. NASA TM X-2672		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle PROCEDURE FOR RAPID DETERMINATION OF NICKEL, COBALT, AND CHROMIUM IN AIRBORNE PARTICULATE SAMPLES				5. Report Date November 1972	
				6. Performing Organization Code	
7. Author(s) Warren F. Davis and Judson W. Graab				8. Performing Organization Report No. E-7105	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135				10. Work Unit No. 501-01	
				11. Contract or Grant No.	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				13. Type of Report and Period Covered Technical Memorandum	
				14. Sponsoring Agency Code	
15. Supplementary Notes					
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17. Key Words (Suggested by Author(s)) Atomic absorption spectroscopy Chromium Spectroscopy Air samples Nickel Analysis Cobalt				18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		22. Price* \$3.00	
				21. No. of Pages 16	

PROCEDURE FOR RAPID DETERMINATION OF NICKEL, COBALT, AND CHROMIUM IN AIRBORNE PARTICULATE SAMPLES

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SUMMARY

This report describes a rapid procedure for the determination of 1 to 20 micrograms of nickel, chromium, and cobalt in airborne particulates collected in the powder-metals laboratory.

Previously used methods for determining trace metals in airborne particulates, such as colorimetric analysis, emission spectroscopy, or neutron activation analysis, were judged either to be too slow or to require instrumentation that was too complex and/or not available. This procedure was developed to satisfy a need for a rapid, selective, and sensitive method for determining nickel, chromium, and cobalt.

The method described utilizes the combined techniques of low-temperature ashing and atomic absorption spectroscopy. The airborne particulates are collected on analytical filter paper by means of diaphragm pumps. The filter papers are quickly and completely ashed at low temperature in a 10-cubic-centimeter beaker in a stream of electronically excited oxygen. The residues are dissolved in hydrochloric acid, and nickel, chromium, and cobalt are determined directly without transfer with good precision and accuracy by means of atomic absorption. Speed and convenience are increased by use of a dual-channel instrument, which permits simultaneous measurement of two elements, and by the use of the same flame conditions for all three elements. Since no separations, filtrations, or transfers of solutions are necessary, 20 samples may be analyzed in 2 hours.

Recovery of weighed amounts of nickel, chromium, and cobalt powders was 92 to 100 percent complete. The effects of flame type, burner height, slit width, and lamp current on the atomic absorption measurements were studied. A moderately rich acetylene-air flame was selected for measurement of the three elements because each exhibits good sensitivity with this condition. Interference effects which may dictate the use of an oxidizing flame for the determination of nickel and cobalt were absent.

INTRODUCTION

A survey (ref. 1) was conducted in 1966 by Viles, Chamberlin, and Boylen to obtain information on the toxicological properties of and to investigate methods for the analytical determination of certain metals and metal oxides used in the Lewis Research Center powder-metals laboratory. The materials studied included the following: nickel, tungsten, thorium oxide, aluminum, aluminum oxide, magnesium oxide, zirconium oxide, niobium, molybdenum, cobalt, and chromium. Special emphasis was placed on the influence that extremely small particle sizes, 0.002 to 0.03 micrometer (20 to 300 Å), may have on the toxic hazard of exposure to many metals or metal oxides.

A lack of information concerning the physical, chemical, and biological behavior of metal particles in the ultrafine size range required that a conservative view be taken when estimating the potential toxicological hazard of these metal powders. The suggested maximum airborne particle concentrations are merely estimates of what is believed would provide reasonable assurance of safety to the health of employees. The report recommended a threshold limit value (TLV) of 0.002 milligram per cubic meter for cobalt, chromium, and nickel. This TLV for nickel as ultrafine powder particles is the same as that recommended in 1966 by the American Conference of Governmental and Industrial Hygienists (ACGIH) for nickel carbonyl. As late as 1971, ACGIH had set no limits for cobalt, chromium, and nickel particles of less than 0.3 micrometer.

Colorimetric or neutron activation methods of analysis were used by Viles, Chamberlin, and Boylen for determining all the metals but thorium, which was measured by its alpha decay rate.

It was concluded (ref. 1, p. 7) that nickel, cobalt, chromium, and thoria were the most hazardous of the materials studied. Nickel, cobalt, and chromium are also among the most widely used metals at the Lewis Research Center, as many high-temperature alloys are based on them. Thoria is dispersed in small amounts in certain alloys to increase strength.

As a result of the recommendations of reference 1, a surveillance program was instituted. Airborne particulate samples were taken at operator positions near ball mills, ultrasonic cleaners, attritors, blenders, dry boxes, and hoods. Samples were also taken at building exhausts. High-efficiency particulate air (HEPA) filters were installed in all exhausts. A HEPA medium has a minimum efficiency rating of 99.97 percent for 0.3-micrometer particles (ref. 2) as determined by the dioctyl phthalate (DOP) test method at airflows of 20 and 100 percent of the rated flow capacity of the filter assembly. Sampling of the exhausts would detect failure of the HEPA filters and possible contamination of the immediate area. Atmospheric contamination outside the building was proven to be negligible.

Methods frequently used for determining trace metals in airborne particulates, such as colorimetric analysis, emission spectroscopy, or neutron activation analysis, were

judged either to be too slow or to require instrumentation that was too complex. The procedure described in the next section, utilizing low-temperature ashing (LTA) and atomic absorption spectroscopy (AAS), has the advantages of speed, selectivity, and sensitivity. AAS has been applied recently to the analysis of toxic metals in samples of airborne particulates (refs. 3 to 6).

Rapid analysis of samples is necessary in order to be able to locate and correct an unsafe condition promptly. In the procedure described, air particulate samples are collected on filter papers and are ashed at low temperature, and the residues are dissolved in acid. After dilution, the solutions are analyzed for nickel, chromium, and cobalt by AAS. Since no separations, filtrations, evaporations, or transfers of solution are necessary, 20 samples may be analyzed for the three elements in 2 hours. The method of analysis is described in the appendix.

EXPERIMENTAL WORK

Sample Collection

As recommended by the survey of Viles, Chamberlin, and Boylen, 2.5-centimeter Whatman 41 filter paper was adopted as the collection medium. Glass-fiber filters contain significant amounts of many elements including nickel and chromium (ref. 3). The high concentration of soluble salts causes a significant matrix effect (ref. 5). Upon treatment with a mixture of hydrochloric and hydrofluoric acids, the resulting semi-dissolved solids were observed to decrease the aspiration rate and sometimes plug the plastic aspiration tubing leading to the nebulizer of the atomic absorption spectrometer. Use of Whatman 41 ashless filter paper eliminates the plugging problem, lowers the blank, and avoids the matrix effect of the glass-fiber filters.

An article by Dams, Rahn, and Winchester (ref. 7) published after this work was completed evaluated various filter materials for suitability for atmospheric particulate sampling and elemental analysis by neutron activation. It was concluded that, of the commercially available filters, Whatman 41 is optimum from the standpoints of low blanks, particle retentivity, and ease of handling.

Samples were collected on 2.5-centimeter Whatman 41 filter papers supported by filter funnels. An airflow was supplied by Universal Electric diaphragm pumps, model 8T-012. The pumps were started and stopped by individual electric timers. Samples were collected only during working hours. The sampling time was 40 hours, and the volume of air, measured by a gas meter, was usually 10 to 20 cubic meters.

Areas where samples were collected included the following: a work bench, an attritor hood, a dry-box air lock, a powder weighing area, four blowers with HEPA fil-

ters, two hydrogen cleaners, a roof vacuum pump exhaust, a hood for radioactive materials, a machine shop grinding area, and three exhaust manifolds.

Sample Ashing

The use of the low-temperature ashing technique for the treatment of air sample filter papers appeared to offer several advantages over conventional wet ashing with mineral acids or dry ashing in a muffle furnace at about 500° C. These include speed, convenience, and less chance of contamination or loss. Acids are a possible source of contamination, and a carbonized residue remains. Ignition in a furnace may cause contamination from the container or atmosphere, and certain elements may be volatilized or converted to refractory compounds. The use of a low-temperature asher (refs. 8 and 9) avoids most of these disadvantages and is faster as well.

In this work, a Tracerlab model LTA-505 low-temperature asher was used to ash the filter paper samples quickly and completely. Five samples can be ashed simultaneously at relatively low temperature by electronically excited oxygen without danger of contamination (ref. 10). In the case of very volatile elements, for example, mercury, selenium, and arsenic, losses may be significant (ref. 8). The asher is illustrated schematically in figure 1.

Oxygen flows into a common inlet manifold, and at reduced pressure and low flow rate, passes down through five separate vertical tubes surrounded by a radiofrequency coil. The coil generates a high-frequency electromagnetic field in which molecular oxygen is converted to excited, very reactive atomic and ionic species. This mixture flows through horizontal oxidation chambers containing the samples.

Volatile products of combustion and excess oxygen are drawn into an exhaust manifold and removed by a vacuum pump. The actual temperature reached by the sample depends on such factors as the thermal conductivity and specific heat of the sample, the concentration of excited oxygen available, and the type of chemical reaction, that is, whether it is exothermic or endothermic. The concentration of excited oxygen is directly controlled by the power input to the radiofrequency coil, and to some extent, by the flow rate and pressure of the molecular oxygen entering the system. Ashing rate is affected by the sample temperature, the amount of surface area exposed, and the inorganic content of the sample. The time required to ash a 2.5-centimeter filter paper disk completely was observed to vary with the amount of particulate matter it contains and also its orientation in the stream of electronically excited oxygen. Oxidation is most rapid when the paper is upright in a 10-cubic-centimeter beaker. Ashing is usually complete in 10 to 15 minutes.

Sample Solution

The metallic residues are heated gently in covered 10-cubic-centimeter beakers with 0.25 cubic centimeter of 12 molar hydrochloric acid for 10 to 15 minutes. Finally, the clear solutions are diluted with 10 cubic centimeters of 1 volume percent hydrochloric acid added by buret. The beakers are covered and warmed briefly to obtain mixing.

Sample Measurement

The concentrations of the three elements in the solutions are determined by atomic absorption spectroscopy. The instrument used to obtain the absorption measurements was an Instrumentation Laboratory model 153 atomic absorption spectrophotometer (ref. 11). This instrument is manufactured with two double-beam optical channels. In one channel (channel A) there is a 1/3 meter Ebert grating monochromator with 1200 lines per millimeter, and in the other (channel B) an interference filter is used for wavelength selection. Nickel and cobalt are determined in channel A at 232.0 and 240.7 nanometers, respectively, and simultaneously, chromium is determined in channel B by using a 357.9-nanometer interference filter monochromator. Single-element high-intensity hollow cathode lamps were used for each element. A Bolying three-slot laminar flow burner with a rich acetylene-air flame was used for all the measurements. Variable scale expansion is available in each channel. The instrument parameters used for the measurements are listed in table I.

After blank corrections are made, the concentrations of nickel, chromium, and cobalt are calculated by comparison with measurements of standard solutions of the metals in 1 volume percent hydrochloric acid.

RESULTS

Recovery Experiment

Experiments were performed to determine the recovery of nickel, chromium, and cobalt by using the procedure described. About 1-milligram amounts of each of the finely divided metals were weighed on a microbalance into 10-cubic-centimeter glass beakers. A 2.5-centimeter Whatman 41 filter paper disk was placed upright in the sample beakers and in blank beakers. The papers were ashed at maximum radiofrequency power with the oxygen flow adjusted to about 50 cubic centimeters per minute. A chamber pressure of about 133 newtons per square meter (1 mmHg) was maintained by the

oxygen flow. Slow pumping rates and pressure changes were used to avoid disturbing the finely divided metals.

The metal residues were dissolved by heating with small amounts of 12 molar hydrochloric acid. The solutions were diluted to 100 cubic centimeters with 1 volume percent hydrochloric acid, and then aliquots were further diluted so that the metals were in the concentration range of about 0.5 to 1.7 ppm. Finally, the recovery of the three metals was determined by comparison of their atomic absorption measurements, using the parameters in table I, with those of standards prepared from the same metals. The results of the recovery experiment are presented in table II. A recovery of 92 to 100 percent is considered satisfactory at this level and gives confidence in the precision and accuracy of the procedure.

Effect of Instrument Parameters on Sensitivity

Calibration curves for nickel, chromium, and cobalt are given in figures 2 to 4. In the cases of nickel and cobalt, the variation of sensitivity with slit width is shown. Since chromium is determined in channel B by using an interference filter, the effect of slit width on this element is not shown. Higher sensitivity is obtained for nickel and cobalt with a 40-micrometer slit width than with a wider setting. However, the main reason for the use of a narrow slit was references in the literature (refs. 11 and 12) to the presence of nonabsorbing lines (231.7, 232.1, and 240.77 nm) close to the nickel and cobalt absorption lines at 232.0 and 240.72 nanometers. The absorption lines used for these two elements are the most sensitive available, but a narrow bandpass is necessary. A decrease in the slit width results in an increase in sensitivity but at the expense of a noisier signal (ref. 11).

The effect of hollow cathode lamp current on the sensitivity of absorption by nickel, chromium, and cobalt is indicated in figure 5. Both nickel and cobalt show decreases in absorption with an increase in lamp current, but chromium absorption is unchanged.

The effects of burner height and type of flame on absorption by nickel, chromium, and cobalt are shown in figures 6 to 8. Burner height is the distance from the top of the Belling burner to the horizontal beam of the hollow cathode lamp. A lean flame and an increase in burner height result in the poorest sensitivity for all three elements. Because chromium forms a stable monoxide (ref. 13), it shows the largest increase in sensitivity as the flame is made richer. Chromium has a threefold higher sensitivity in a moderately rich flame than in a lean flame. A burner height of 10 millimeters and a slightly luminous flame was adopted for all measurements as this was convenient and nearly optimum for all three elements. The desired degree of luminosity can be reproduced more easily by visual observation than by a fuel-air pressure ratio. In these

samples, significant interferences are absent. Generally, however, an oxidizing non-luminous flame is used for nickel and cobalt (ref. 11).

Size and Variation of Blank

Blank determinations are made by taking 2.5-centimeter Whatman 41 filter paper disks through the ashing, dissolution, and measurement steps exactly as for samples. A total of 12 blank determinations performed on 5 days, calculated to the equivalent amount of nickel, chromium, and cobalt, were as follows:

	Nickel	Chromium	Cobalt
Average blank, μg	0.18	0.03	0.06
Standard deviation, μg	.09	.08	.06

CONCLUDING REMARKS

A relatively simple and rapid procedure has been described for the determination of 1 to 20 micrograms of nickel, chromium, and cobalt in airborne particulate samples. The procedure combines the advantages of LTA and AAS and requires a minimum amount of handling.

The use of these techniques makes possible the analysis of 20 samples in 2 hours. If any of the three elements is found to exceed the TLV at any station, appropriate action may be taken quickly. The lack of interferences and high sensitivity make possible the measurement of all three elements in one solution with a single set of flame conditions.

Use of analytical grade filter paper for sampling and the low-temperature asher for oxidation requires less time and less acids than wet ashing. Also, there is less chance of contamination, volatilization loss, or conversion to refractory compounds than in ashing in a muffle furnace at 500^o to 550^o C.

The use of filter paper and the LTA instead of glass-fiber filters and acid leaching results in a clear solution with a low blank, no carbonaceous residue requiring separation, and no semi-dissolved solids to cause a matrix effect and plugging of the aspirator tubing.

The recovery of nickel, chromium, and cobalt metals is 92 to 100 percent complete. This is considered satisfactory in the 0.2 to 1.0 ppm range and gives confidence in the precision and accuracy of the procedure.

Variation of sensitivity of the three metals as a function of slit width, lamp current, burner height, and flame type was studied. A moderately rich acetylene-air flame was selected to optimize sensitivity for chromium, but it was also found very satisfactory for nickel and cobalt since interferences were absent. Sensitivity decreases slightly with increasing burner height. Nickel and cobalt show decreasing sensitivity with increasing lamp current, but the sensitivity of chromium is constant. Sensitivity of nickel and cobalt in channel A is an inverse function of slit width. Use of a narrow slit is recommended because of nonabsorbing lines close to the most sensitive nickel and cobalt lines. This results in a slightly noisier signal, but calibration curves are linear. Calibration with standards is done with each set of samples to compensate for slight variations in instrument operating conditions.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 14, 1972,
501-01.

APPENDIX - METHOD OF ANALYSIS

The following equipment and chemicals are used in the analysis:

- (1) Atomic absorption spectrophotometer with two double-beam optical channels
- (2) Low-temperature asher
- (3) Diaphragm pumps
- (4) Ashless filter paper
- (5) Stock solutions, 10 000 ppm of nickel, chromium, and cobalt
- (6) Hydrochloric acid, 12 molar and 1 volume percent dilution

The procedure is as follows:

(1) Air filter samples and blank filter papers are placed upright in 10-cubic-centimeter glass beakers. These beakers are placed in trays in the individual chambers of the LTA. The LTA is operated according to the manufacturer's recommendations.

(2) After ashing, 0.2 to 0.4 cubic centimeter of 12 molar hydrochloric acid is added to the residues in the beakers, and the beakers are covered and heated briefly (15 to 20 min).

(3) Then 10 cubic centimeters of 1 volume percent hydrochloric acid is added to the beakers, and they are covered and warmed for approximately 10 minutes to obtain mixing.

(4) Atomic absorption measurements of nickel, chromium, and cobalt are made by using the parameters listed in table I.

(5) Standards and blanks are run with each set of samples to compensate for slight changes in sensitivity resulting from not exactly reproducing the desired instrument conditions. After blank corrections are made, concentrations of the three elements are calculated by comparison with measurements of standard solutions of the metals diluted with 1 volume percent hydrochloric acid. Results are calculated as milligrams of metal per cubic meter of air sampled.

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TABLE I. - INSTRUMENT CONDITIONS

Burner	Boling
Burner height, mm	10
Detector 1P28 voltage, V	700
Hollow cathode lamp current, mA	8
Slit width, μm	40
Aspiration rate, cm^3/min	4
Acetylene pressure, N/cm^2 (lb/in. ²)	3.8 (5.5)
Air pressure, N/cm^2 (lb/in. ²)	3.4 (5.0)
Scale expansion	Factor of 5 or 10
Integration period, sec	10
Wavelength, nm	
Nickel	232.0
Cobalt	240.7
Chromium (filter)	357.9

TABLE II. - RECOVERY OF NICKEL,
CHROMIUM, AND COBALT AFTER
LOW-TEMPERATURE ASHING

Metal	Weight, mg	Quantity added, ^a ppm	Quantity found, ppm	Recovery, percent
Nickel	1.010	0.50	0.49	98.0
		1.01	.98	97.0
	1.100	0.55	0.53	96.4
		1.10	1.01	91.8
Chromium	1.720	0.86	0.80	93.0
		1.72	1.60	93.0
	0.960	0.48	0.46	95.8
		.96	.92	95.8
Cobalt	0.580	0.58	0.55	95.0
		.58	.57	98.6
	0.775	0.775	0.77	99.2
		.775	.77	98.8

^aDissolved metal powders were diluted to 100 cm^3 with 1 vol. % hydrochloric acid. Then aliquots were diluted further to match concentration range of nickel, chromium, and cobalt in air samples.

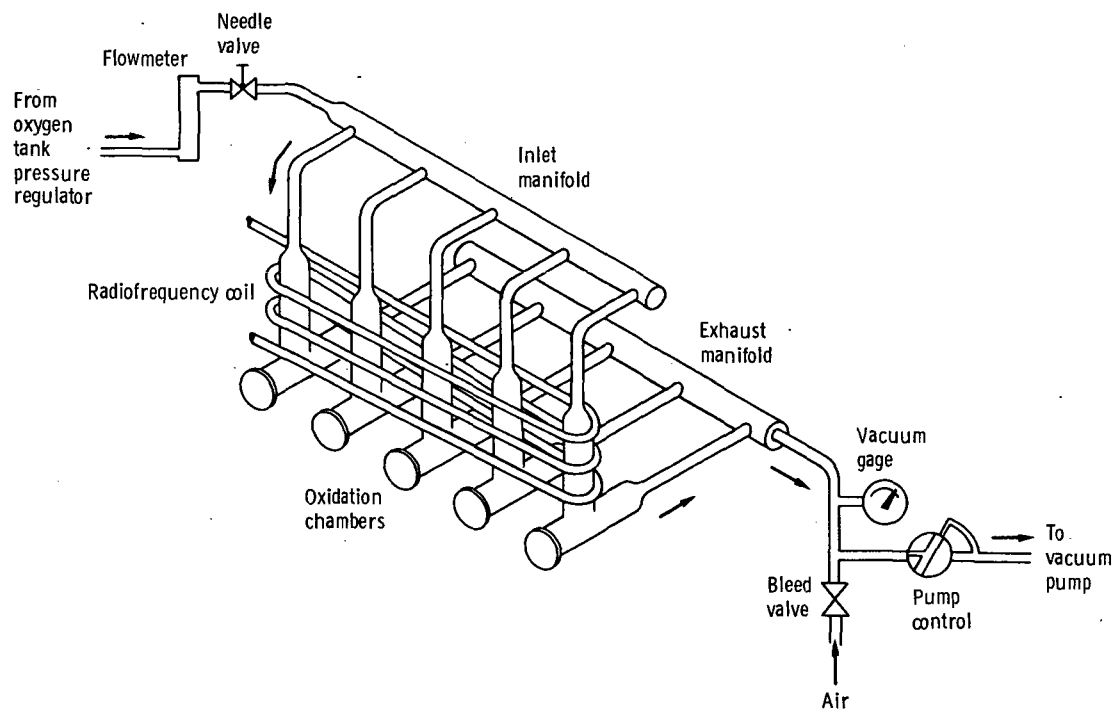


Figure 1. - Schematic diagram of low-temperature asher.

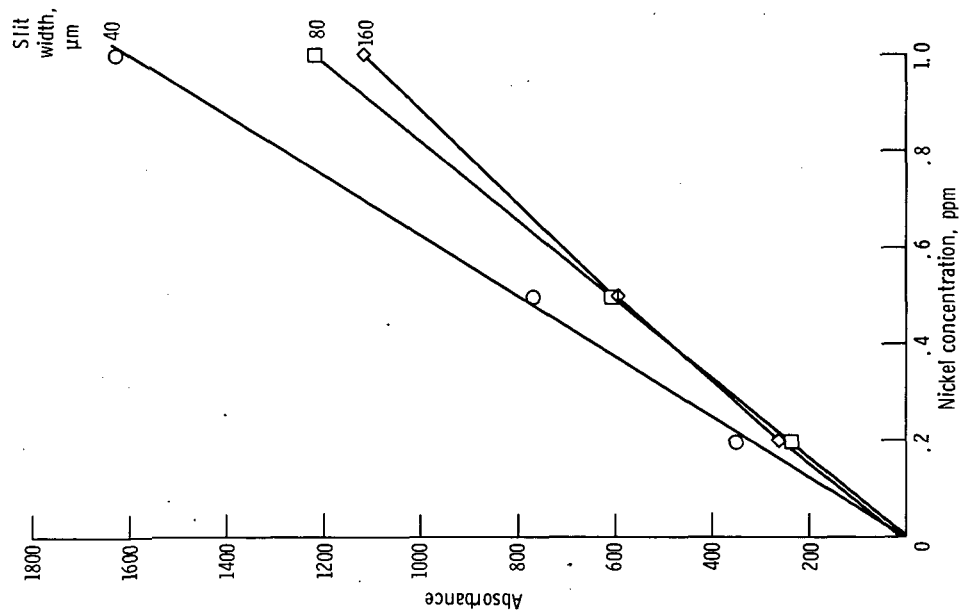


Figure 2. - Nickel calibration curve at varying slit widths. Acid medium, 1 volume percent hydrochloric acid.

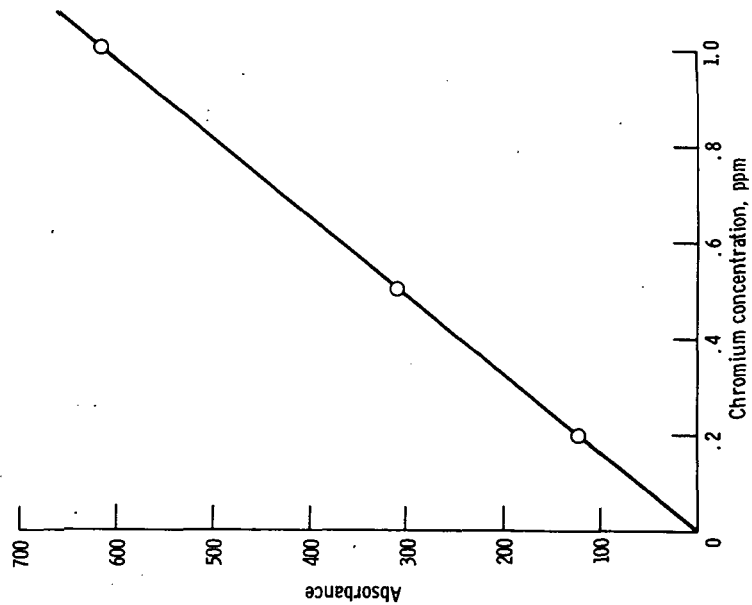


Figure 3. - Chromium calibration curve, determined in channel B with interference filter. Acid medium, 1 volume percent hydrochloric acid.

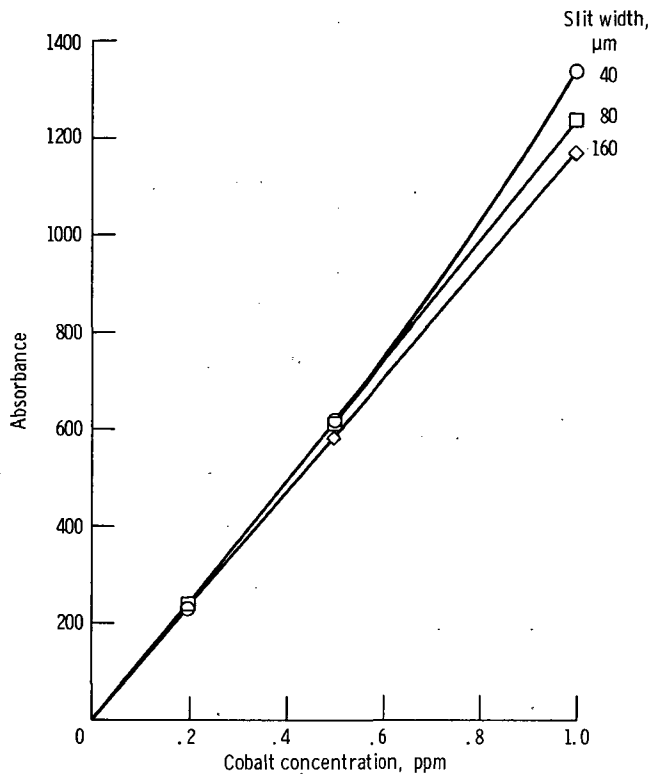


Figure 4. - Cobalt calibration curve at varying slit widths. Acid medium, 1 volume percent hydrochloric acid.

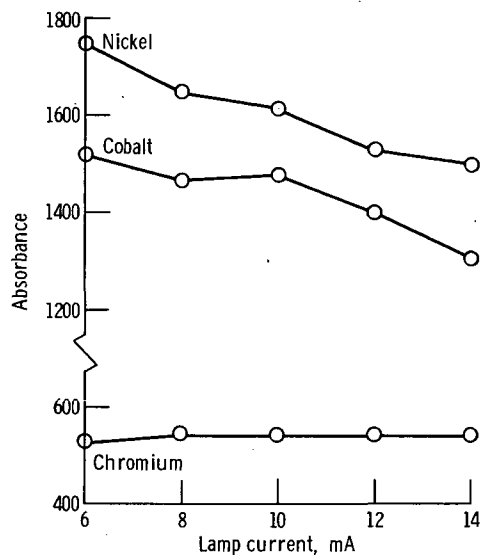


Figure 5. - Nickel, chromium, and cobalt absorption at varying lamp currents. Acid medium, 1 volume percent hydrochloric acid.

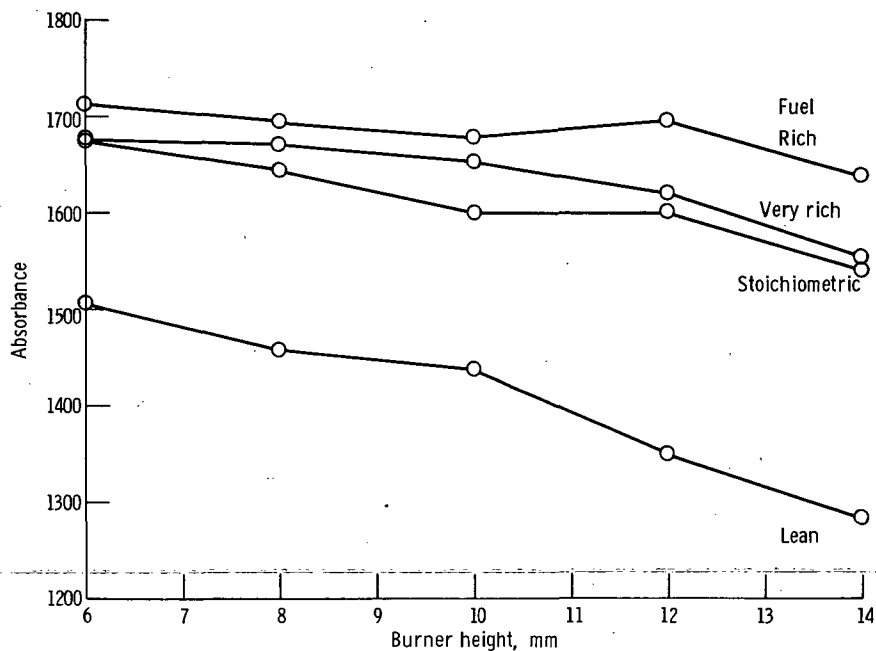


Figure 6. - Nickel absorption as function of flame type and burner height. Acid medium, 1 volume percent hydrochloric acid; nickel concentration, 1 ppm.

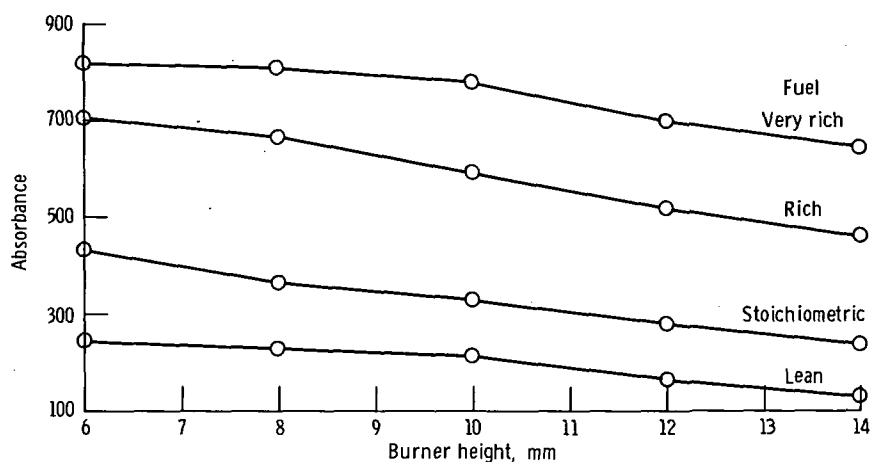


Figure 7. - Chromium absorption as function of flame type and burner height. Acid medium, 1 volume percent hydrochloric acid; chromium concentration, 1 ppm.

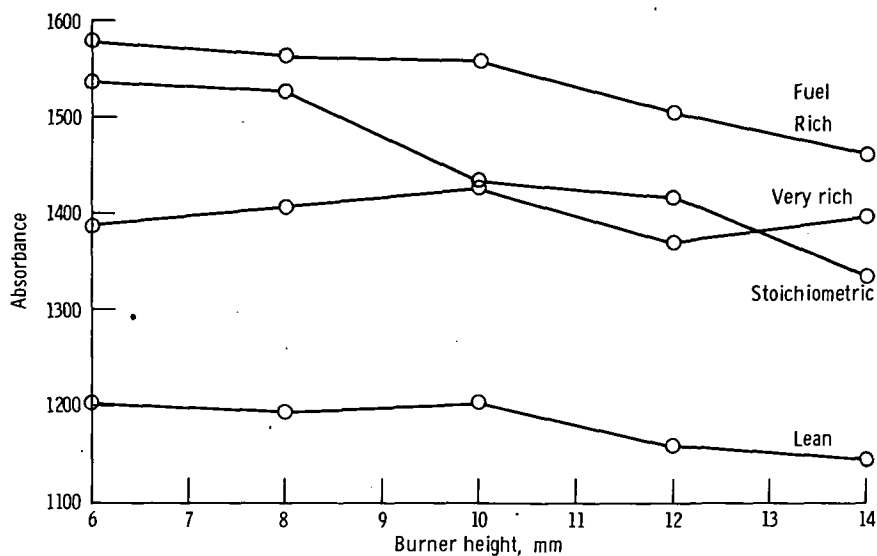


Figure 8. - Cobalt absorption as function of flame type and burner height. Acid medium, 1 volume percent hydrochloric acid; cobalt concentration, 1 ppm.



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